# This Page Is Inserted by IFW Operations and is not a part of the Official Record

### BEST AVAILABLE IMAGES

Defective images within this document are accurate representation of The original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

## IMAGES ARE BEST AVAILABLE COPY.

As rescanning documents will not correct images, please do not report the images to the Image Problem Mailbox.

Q

JP-4-70905

BD

- (19) Japan Patent Office (JP)
- (12) Public Report of Patent (B2)

distinguishing mark 300

office reference number

8822-4D

9052-4C 8823-4D

7199-3B

- (11) Announcement of Patent Application
- (24) (44) Announcement

Nov. 12, 1992

Number of Invention

1 (total of 3 pages)

(54) Name of Invention

Pasteurized Porous Polyolefin Hollow Fiber

(21) Special Application

Showa59-130557

(22) Application

June 25,1984

(23) Exhibition

Showa61-8104

(43) Jan. 14, 1986

(72) Inventor

Saburo Hiraoka

Mitsubishi Rayon Co.Ltd. 610 Daikou-chou . Nagoya, Aichi

(72) Inventor

Shouichi Nagai

Mitsubishi Rayon Co.Ltd. 610 Daikou-chou Nagoya, Aichi

(72) Inventor

Mitsuo Chiga

Mitsubishi Rayon Co.Ltd. 610 Daikou-chou Nagoya, Aichi

(71) Applicant

ره. ٣

Mit ubishi Rayon Co.Ltd.

2-3-19 Kyoubashi Chuo-ku, Tokyo

Examiner

Tomo Hattori

#### (57) Range of the patent rights claimed

1 Pasteurized porous polyolefin which is characterized as follows;

A micro-hollow ranges from the internal surface of the hollow fiber to the external surface, and vice versa, and waterinsoluble porous acrylonitrile polymer thin-film containing silver is deposited on the surface of the laminating structure of the porous polyolefin hollow fiber having a laminating structure.

Detailed explanation of the invention

[Field of the technical use]

The present invention pertains to the porous polyolefin hollow fiber having excellent pasteurization.

[Background techniques]

Conventionally, various types of devices have been proposed as a device for manufacturing pure water that is used in the medical field, pharmaceutical field, food industries, precision engineering field, and for the scientific and chemical experiments.

That is, there are a device for manufacturing pure water which does not contain colloid materials or bacteria, a device for manufacturing pure water which does not contain exothermic materials, and a device for manufacturing pure water which does not contain radioactive materials. For example, devices for manufacturing sterile water used in the pharmaceutical field includes a device using distillation, a boiling-sterilization device, and a pasteurization device using ultraviolet rays. However, the energy costs and manufacturing costs are high, and the sterilization may not be sufficient. Moreover, exothermic materials (pyrodiene) may not be removed although colloid materials and bacteria can be removed.

In order to solve the above problem, a reverse osmosis film device has been adopted, however, the device requires high pressure, resulting in high energy costs and manufacturing costs.

Considering the situation, the applicant of the present invention proposed a porous polyolefin hollow fiber having a laminating

structure where a micro hollow ranges from the internal surface of the hollow fiber to the external surface, and vice versa, as a porous hollow fiber which solves the problem that the conventional devices have, has low manufacturing costs and energy costs, and simple structure with little trouble, and is suitable for the highly reliable precise-filtering device. However, although the porous polyolefin hollow fiber prevents bacteria from penetrating, unfiltrated bacteria may gradually propagate, and accumulate inside the module using this fiber, for the fiber itself does not have the pasteurization nature. This is sanitarily unfavorable, and a porous polyolefin hollow fiber with excellent pasteurization had been desired.

#### [Purpose of the invention]

The purpose of the present invention is to provide a pasteurized porous polyolefin hollow fiber.

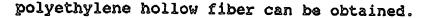
#### [Structure of the invention]

The present invention pertains to the pasteurized porous polyolefin hollow fiber which is characterized by the fact that a micro hollow ranges from the internal surface to the external surface of the hollow fiber, and that water-insoluble porous acrylonitrile polymer thin-film containing silver is deposited on the surface of the laminating structure of the porous polyolefin hollow fiber. Polyethylene, polypropylene, and polytetrafluoroethylene can be used as polyolefin. Taking polyethylene as an example, a porous polyethylene hollow fiber having a laminating structure where a micro hollow ranges from the internal surface to the external surface of the hollow fiber can be obtained according to Patent Application Showa55-116263.

As a method to deposit the water-insoluble porous acrylonitrile polymer thin-film containing silver on the surface of the laminating structure of the porous polyethylene hollow fiber to obtain a permanent pasteurized porous polyethylene hollow fiber, a water-insoluble acrylonitrile porous film can be formed firmly by soaking the porous polyethylene hollow fiber in the water which is a coagulant, after the porous polyethylene hollow fiber contains the organic solvent solution containing acrylonitrile polymer with a water-insoluble acid group.

Then, the porous polyethylene hollow fiber where the acrylonitrile porous film containing the water-insoluble group is deposited is soaked in the solution containing silver nitrate in order to obtain the porous polyethylene hollow fiber where silver ions are adsorbed in the acid group in the acrylonitrile porous film.

After that this fiber is processed in the reducing agent to form a m tallic silver, and a hollow fiber where a water-insoluble porous acrylonitril polym r film containing silver is deposited on the surface of the laminating structure of the porous



Silv r shows exc llent bacteria-natur (translator's note: misspelled term? should be pasteurization-nature???), and if an appropriate method is used, it is possibl that silver can purify water having approximately 10,000,000 multiplication of its weight. It is known that water becomes sterile when it is placed in a silver container.

Therefore, if a small amount of silver is deposited on the surface of the laminating structure of the porous polyolefin hollow fiber, excellent pasteurization nature can be obtained, similar to the case where silver containers are used.

A module can be made using the pasteurized porous polyolefin hollow fiber of the present invention by applying a general module manufacturing method using a porous hollow fiber, and any module where liquid or air can be filtrated from the external surface to the internal surface, or from the internal surface to the external surface of the hollow fiber is sufficient.

The pasteurized porous polyolefin hollow fiber of the present invention enables the removal the colloid materials, bacteria, and exothermic materials as well as the highly reliable precise filtration with lower manufacturing costs and energy costs, and simple structure with little trouble, compared with the conventional devices, for the unfiltrated bacteria remaining on the surface of the module are pasteurized by silver, so the module is maintained safely and sanitarily.

#### [Examples]

Following is an explanation of the present invention, referring to the examples. Following method was used for the measurement of the pasteurization.

#### (Measurement of the pasteurization)

A test piece was placed on the agar culture medium where yellow staphylococcus was planted, and bacteria were incubated at 37 degrees C for 24 hours to evaluate the pasteurization based on the existence of the staphylococcus around the test piece.

#### (Evaluation)

- o: No bacteria growth is recognized around the test piece, and hollow occurs.
- $\Delta$  : No bacteria growth is recognized around the test piece, and no hollow occurs.
- x : Bacteria growth is recognized around the test piece.

#### Example 1

After soaking a porous polyethylene hollow fiber EHF (product name, manufactured by Mitsubishi Rayon, Co.Ltd.) in the solution obtained by dissolving acrylonitrile polymer 0.5 weight-parts consisting of acrylonitrile 93 weight-%, vinyl acetate 7 weight-%, sulfonic acid group 50m.mol/kg. polymer with dimethylformamide 99.5 weight-parts at 25 degrees C, the solution was drained to realize the deposition of the solution of 220% owf on the hollow fiber . Next, the hollow fiber was soaked in 60-degree-C hot water to make the hollow fiber porous, and to eliminate the solution due to a rapid solidification of the acrylonitrile polymer. After that the hollow fiber was washed well with water, and we obtained a porous polyethylene hollow fiber where an acrylonitrile porous film was deposited on the surface of laminating structure of the hollow fiber. Then, after the hollow fiber was soaked in the solution containing silver nitrate 1 weight-% at 25 degrees C to bond the silver ions with the acid group in the acrylonitrile porous film, the hollow fiber was soaked in the solution containing hydrazine hydrate 1 weight-% at 25 degrees C for reduction processing. After that, the hollow fiber was taken out, was washed with water, and was dried by using a 60-degree C hot-air dryer. As a result, we obtained a hollow fiber where a water-insoluble porous acrylonitrile polymer film containing silver was deposited on the surface of the laminating structure of the porous polyethylene hollow fiber. We created a module whose resin part is 4cm-long, and whose effective length of the hollow fiber is 10cm by placing 100 hollow fibers in a U-shape, and by fixing the open parts of the hollow fibers with resin.

When the water-penetrating speed was measured under the pressure of 380mmHg at the external surface of the hollow fibers using the above module, the decrease in the water-penetrating speed was extremely low, compared with the module created in the same way using porous polyethylene hollow fibers that were not processed in the manner described above.

Furthermore, we took out hollow fibers to evaluate the relationship between the amount of water-penetration and pasteurization for yellow staphylococcus. The result is shown below.

	amount of water penetration	pasteurization (yallow staphylo- coccus)
present invention	0 5 10	0 0
comparison example	0	х

It is clear that the porous polyethylene hollow fiber of the

present invention wh r porous acrylonitrile polymer film containing silver was deposited has excell nt p rman nt pasteurization nature although the conventional porous polyethylen hollow fib r shown as a comparison example do s not have pasteurization. It is also clear that even when 10 l of water was filtrated, no bacteria growth was recognized around th test piece, and hollow occurred, showing excellent pasteurization.

10 1200771

The second of th